

TITLE: CHEMICAL AND GEOLOGIC SEQUESTRATION OF CARBON DIOXIDE

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DESCRIPTION: According to the DOE report Carbon Sequestration Research and Development, “Geologic formations, such as oil fields, coal-beds, and aquifers, are likely to provide the first large-scale opportunity for concentrated sequestration of CO₂” [1]. The fossil fuel community has substantial experience injecting CO₂ into geological formations for enhanced oil and gas recovery, enhanced methane recovery from coal-seams, and other applications. Thus, sequestration of CO₂ in some geologic formations may also be accompanied by production of increased amounts of gas or oil, offsetting some of the sequestration costs.

However, there are significant fundamental research needs that must be addressed before geologic formations can be widely used for carbon sequestration. These needs have been identified in the DOE report Working Paper on Carbon Sequestration Science and Technology [2] and include the following: (1) understanding fluid-flow phenomena, CO₂ transport, and the roles of fracture networks and chemical changes, which affect the sequestration efficiency of CO₂, (2) identifying optimal sites for CO₂ sequestration, (3) evaluating the integrity of injection strata and any short-term environmental effects of CO₂ sequestration, and (4) developing methods to quantify and verify the amount of CO₂ sequestered.

RESEARCH OBJECTIVES: The near term objectives of the geological sequestration task include the following: (1) Make major advances in the development of universal, physical descriptions of immiscible flow using measurements to validate the flow models, (2) develop measurement of fluid-flow through fractures using high-resolution computerized tomographic (CT) imaging and modeling of flow through fractures, (3) using synthetic and field brines, investigate the solution kinetics of reactions of CO₂ in brine and the reactivity of various mineral substrates and construct a supporting database, (4) for CO₂ sequestration in coal-seams, perform (a) reservoir simulations and (b) measurements, such as mass and heat of CO₂ sorption/CH₄ desorption, (5) develop a comprehensive monitoring program to verify the integrity of geologically sequestered CO₂ in a variety of geologic formations, including use of tracer molecules and a monitoring protocol, and (6) design a geologic sequestration flow laboratory.

LONG TERM GOALS: The vision described in Carbon Sequestration Research and Development states, “By the year 2025, effective, safe, and cost competitive options for geological sequestration of all the CO₂ generated from coal, oil, and gas power plants and generated by H₂ production from fossil fuels will be available within 500 km of each power plant.” [1]. The long term goal of the OST geological sequestration program is to develop the scientific and technological aspects of geological sequestration to the point where that vision comes to fruition.

SUMMARY ACCOMPLISHMENTS:

MODELING FOR SEQUESTRATION IN BRINE FIELDS, OIL AND GAS FIELDS, AND NATURAL GAS HYDRATES

Eight papers were published in the conference proceedings of the First National Conference on Carbon Sequestration, Washington, DC, May 15-17, 2001 and the ICCS/IEA Meeting, San Francisco, CA. (The latter meeting was cancelled, but the proceedings were published.) Several other papers were submitted for journal publication.

Two-Phase Flow Through Porous Media

Rule-based modeling calculations of gas-water flow through porous media were performed and used to describe a residual-saturation surface as a function of viscous, capillary, and buoyancy forces. The design, writing, and testing of a network model was completed. This model is much more general than the diffusion-limited aggregation (DLA) or invasion percolation with trapping (IPt) models, but gave results consistent with these widely accepted models for their special conditions. Experiments on two-phase flow through porous media measured flow patterns, saturations, relative permeabilities, and fractal dimensions, all for injection of gas into brine with horizontal flow. The results of the network model of two-phase flow through porous media were compared with the laboratory measurements. The beginning of the transition from IPt behavior towards conventional fractional-flow behavior was found as a function of capillary number and volume of fluid injected. Experimental residual saturations as a function of capillary number were extrapolated to predict the capillary number at which the residual saturation of the displaced phase (e.g., brine displaced by gas) would reach zero.

Fracturing And Flow Through Fractures

Berea sandstone cores were fractured using a modified Brazilian test to produce a longitudinal plane fracture. In partnership with Pennsylvania State University, porosity and single-phase flow patterns in the fracture zone were measured using conventional and high-resolution industrial CT imaging. The FLUENT[®] code was used to model single-phase flow through fractures.

Quantitative, Predictive Geology

To initiate a long-term program of quantitative, predictive geology for sedimentary strata, three types of modeling were performed: geostatistical and fractal descriptions of entire river basins, meandering of individual rivers, and sediment transport and deposition in meandering rivers.

Natural Gas Hydrates

A model was developed to interpret experimental data for the effects of pore-size distributions in equilibrium pressure-temperature measurements of clathrate hydrates of methane, ethane, and propane, as well as of carbon dioxide. The method also gives the enthalpy of formation or dissociation for these hydrates. The data and interpretations were extended to the heat of methane/carbon dioxide hydrates, as might occur in sequestration by conversion of naturally occurring methane hydrate.

Web Site

A web site was designed and implemented for the addition of a bibliography, abstracts, and experimental data.

AN INVESTIGATION OF GAS-WATER-ROCK INTERACTIONS AND CHEMISTRY

Analysis of Aqueous Phase Reaction Products (NETL Brine Chemistry Lab):

- Facilities to conduct hydrothermal CO₂-water-rock reactions and analyze these complex mixtures have been developed at NETL. Key instruments have been purchased and are currently being installed.
- A comprehensive literature search was conducted on the current state of knowledge of CO₂ sequestration in deep saline aquifers; the solubility of CO₂ in water, NaCl, and brine; and the thermodynamics of the PVTX relations of the CO₂-H₂O-NaCl system in general.

Experimental Investigation of CO₂ Sequestration in Deep Aquifers (USGS Hydrothermal Laboratory):

- Reactor designs and the CO₂ charging system were improved; improved reactor design to minimize experimental changes during sampling; and separator for controlling CO₂ pressure while charging experiments and sampling was designed and fabricated.
- Sampling and analytical protocol for fluids with high CO₂ partial pressures was developed.
- Experimental design was validated by carrying out experiments in pure water where CO₂ solubility is well established and by measuring CO₂ solubility in natural brine from Paradox Valley.
- Work on systematic study of CO₂ solubility in increasingly complex salt solutions is currently underway.
- Presentation on preliminary results, including experimental design, was made at a special session of the Spring 2001 American Geophysical Union (AGU) Meeting in Boston, May 29 - June 2, 2001.

Construction of a Comprehensive Brine Database: Quantitative and Statistical Evaluation of a Compiled Oil/Gas Brine Field Database.

- Brine mapping study performed by TBEG was extensively reviewed by NETL researchers and an additional seven hundred well data sets added. Also, the extensive brine database tabulated by the USGS, which includes data on over 60,000 brines, has been given to NETL for inclusion in the comprehensive database. This material, combined with the large brine database NETL had already constructed, contains information on most of the formations within the conterminous U.S.
- The information is being added to both an MS Excel® database and ArcView® database. The ArcView® database only includes data on brines for which a spacial location (latitude and longitude) is specified. The Excel® database also contains information on brines where only general location information, such as the state or the name of the formation, is available from the literature.
- Where water chemistries, pH, location, depth, and temperature were given, data was entered in a statistical program, and statistical analyses of brine chemistries were initiated. Using this expanded database, major ions were grouped together as a PIDS (principal ions dissolved solids), which account for 98–99% of total dissolved solids, and this was used to calculate fluid pressure gradient.
- Contacts were made with other sources to inquire if additional brine formation data were available. Pennsylvania Geologic Survey, Oil and Gas Division, provided additional publications and hand-written tables not included in any other databases.

- State Geologic Surveys were contacted to acquire contacts in the oil and gas industry for collection of brines. USGS also provided contacts for oil well brines in the Frio Basin in Texas and the Williston Basin in North Dakota.

Collection of Brines and Surrounding Strata in Locations of Low Seismic Activity Near Power Plants.

- Composite map depicting U.S. power plants, saline formations, and seismic activity probabilities has been constructed.
- Brines were collected from Williston, Oriskany, and Oklahoma basins. Arrangements were made to obtain core samples from these basins and contacts made to collect additional brines.

USGS Modeling Laboratory

- Several codes that compute chemical equilibrium in geologic systems were evaluated for use in modeling CO₂ laboratory and field experiments. For homogeneous systems, codes evaluated were: **SOLMINEQ**[®], **SOLVEQ**[®] (Univ. Oregon), **EQ3**[®] (LLNL), and **REACT**[®] (Geochemist's Workbench, Rockware Inc.). For heterogeneous systems, codes evaluated were: **PATHARC**[®] (Alberta Research Council), **CHILLER**[®] (Univ. Oregon), **EQ6**[®] (LLNL), and **REACT**[®].
- Numerous test cases were run using expected mineral assemblages and fluid compositions which are well known. Considering only thermodynamic equilibrium, all of the codes produced similar results with respect to major elements. However, it is clear that certain data produce results that more closely approach real systems.
- **PATHARC**[®] and **PHREEQC**[®] (for heterogeneous systems including aqueous, mineral, and gas phases) were selected to run with **SOLMINEQ**[®] (computations in homogeneous systems with the aqueous phase only),
- Bill Gunter and Ernie Perkins (authors of **PATHARC**[®]) agreed to work with us to develop a combined software package that is both versatile and comprehensive and that would be available in the public domain. See Perkins et al. for a discussion and comparison of geochemical codes.

University of Pittsburgh Modeling Laboratory

- Presentation entitled "A Tale of Two Rates: Implications of the Discrepancy between Laboratory and Field Feldspar Dissolution Rates on Geological Carbon Sequestration" was presented at the Geological Society of America Annual Meeting & Exposition, November 5-8, 2001 in Boston, MA.

PHYSICS AND CHEMISTRY OF COAL-SEAM CO₂ SEQUESTRATION AND COAL BED METHANE PRODUCTION

Advanced Physical Chemical Concepts Applicable to Both Coal-Seam CO₂ Sequestration and Coal Bed Methane Production

- A method for simultaneously accounting for heats of CO₂ and CH₄ sorption/desorption, moles of CO₂ and CH₄ sorbed/desorbed, extents of dehydration, and sample temperature was developed in the context of concurrent CO₂ sequestration and methane production by coal.
- A manuscript was prepared and accepted for presentation at the 11th International Conference on Coal Science scheduled for September 30-October 5, 2001 in San Francisco, CA.

- It was discovered that methane production levels for coals are relatively high whenever the molar heat of CO₂ sorption is more negative than a certain value, ΔH^* , which falls in the range of -36 to -52 kJ/mole.
- In spite of their high water contents, low-rank coals were identified as potentially useful for CO₂ sequestration and the concurrent production of methane. Moreover, lignites were determined to be potentially better performers than subbituminous coals in this context. This is based on the finding that Beulah-Zap lignite has a relatively high molar heat of CO₂ sorption, dehydrates more rapidly than Wyodak subbituminous coal, and produces methane relatively well both when dry and while in the process of being dehydrated, even at a hypothetical extent of dehydration as low as 37%.
- Mathematical methods for resolving complex calorimetric thermograms containing dehydration endotherms, CO₂/CH₄ sorption exotherms, and CO₂/CH₄ desorption endotherms were developed.

Sorption, Transport, and Environmental Chemistry Relevant to Coal-Seam CO₂ Sequestration

- A static system for the measurement of adsorption isotherms was assembled, pressure-tested, and successfully employed to generate data.
- The static system was used to study the adsorption of gaseous CO₂ onto five of the Argonne premium coals,
- A selection of Argonne premium coals was treated with dilute sulfuric acid and dilute sodium hydroxide to provide a suite of pH-modified coals (at pH 2, 7, and 10).
- Isotherms relating the amount of gaseous CO₂ adsorbed were measured for the 5 Argonne coals.
- Isotherms were generated at 4 temperatures in the temperature range of interest to geologic sequestration in coal seams (22-55 °C).
- An equation which provides an excellent fit to the data sets was derived.
- The derived equation was used to separate the actual surface adsorption from the effects of coal swelling on the isotherm shape.
- The extent of surface adsorption data was fit to 4 adsorption isotherm models in the literature (the Langmuir monolayer, the BET multi-layer, and two Dubinin volume-filling equations). The BET isotherm model was found to give a significantly poorer fit than the other models.
- The extent of actual physical adsorption (moles of CO₂ per gram of coal) was determined from the 3 well-fitting equations, and the values were found to agree within 10% of each other.
- Heats of adsorption were calculated using the temperature dependence of the isotherms and the Clausius-Clapeyron equation.
- Heats of adsorption were also calculated using the Dubinin-Radushkevich and Dubinin-Astakhov equations, and they compared favorably with the Clausius-Clapeyron values.
- Under supercritical conditions, CO₂ was found to extract sufficient material from the coal that lines clogged. Similar behavior during sequestration could lead to changes in permeability.
- The results were presented at the First National Conference on Carbon Sequestration and accepted for presentation at the International Conference on Coal Science and the AIChE National Meeting in 2001.
- Manuscripts describing the work were prepared for submission to peer-reviewed journals.
- Contacts were made at the Pennsylvania State University, the Illinois State Geological Survey, and Consol, with whom collaboration is being pursued.
- A geologic strata simulation apparatus (available from another project) was successfully pressure tested, and a 3.5" diameter x 6" long core sample was received from the Illinois State Geological

Survey.

DEVELOPMENT OF COMPREHENSIVE MONITORING TECHNIQUES FOR VERIFYING THE INTEGRITY OF GEOLOGICALLY SEQUESTERED CARBON DIOXIDE

Following the completion of the project proposal, the merit review process was completed. The project proposal was modified to include suggestions made by the merit reviewers. The project received the highest rating and was selected for presentation at Duquesne University (with a view towards future cooperative agreements) and for the National Laboratory Review Meeting in February. Budget cuts were made in lieu of the postponement of part of the work until FY03 and the elimination of the San Juan basin study.

Background work was completed for the New Mexico site in preparation for carbon dioxide injection in February. The Ophir Corporation issued a report on their background survey conducted in August. In addition to low levels of methane and nondetectable levels of ethane, they isolated a buried leaking petroleum transport line as a methane source. This line is being repaired to prevent interference with monitoring studies. CAT samples (sorbent packets for PFT's) from the background study of last August were analyzed at BNL's Tracer Technology Center. Four packets were exposed under passive sampling conditions for 47 days, and a fifth sample was exposed for 14 hours under active sampling conditions. Background amounts of PFT's in the monitoring holes were found that corresponded to atmospheric levels at the site. Significant levels of hydrocarbons were found, which will require some modification of the sampling procedure for passive CAT's. A detailed monitoring protocol has been completed for the New Mexico monitoring study. It includes the design for active and passive grids, logging procedures, personnel assignments, and various experiments designed to evaluate methods of detecting and reducing atmospheric contamination. Also included are stringent requirements, based upon BNL experience, to prevent cross contamination between the tracer loading and monitoring operations.

Nine identical air circulation pumps were constructed for use in an active sampling grid at a depleted oil well site in New Mexico. This grid will be superimposed on a larger passive grid. The pumps are rugged, provide uniform flow rates, can be buried, and can be exposed to a wide range of temperatures and environments without effecting performance. The casings were tested for holding a vacuum. The pumps were each fabricated for less than \$100 in materials and operate on only 80 milliamps for stand alone operation. Solar cell mounts attached to the monitoring hole pipes were constructed for stand alone pump operation. Other equipment was designed and constructed to meet the unique requirements for tracer monitoring at the New Mexico site. These included an extension driving anvil that allows the use of the Bushhammer with 10 foot sections of monitoring hole pipe while eliminating the need for a ladder. This will also increase the safety of the monitoring hole placement operation.

From our experience last August, we realized that the sandy soil at the New Mexico site would present unique challenges to the design of a jack to remove the pipes once the monitoring work is completed. The semi-fluid soil requires high levels of jacking pressure due to a quicksand effect and

generates ground motion under the jacking pad that must be compensated for. Two very different jacks were designed, constructed, and tested. The more useful of the two jacks was selected for use at the New Mexico site.

SCOPING EXERCISE FOR A GEOLOGIC SEQUESTRATION FLOW LABORATORY

A plan for equipment acquisitions and initial experiments was developed and described in the FY02 workplan and review presentation. This plan was based on literature reviews, visits to other laboratories, and an analysis of program needs and currently available equipment. The planned laboratory would include equipment for performing flow experiments through various geologic sequestration media, including an X-ray diffractometer, a petrographic microscope, a gas chromatograph, an atomic absorption system, a scanning electron microscope, a laser scanning confocal microscope, a solid state and magnetic resonance imaging system, and an X-ray CT scanner. This equipment would allow imaging of geologic samples (including coal, sandstone, etc.) before, during, and after the single-phase or immiscible flow of carbon dioxide, methane, brines, and/or other fluids through the samples, with measurement of the time-dependent composition of the effluent, as well as measurements of the time and spatial-dependence of fluid compositions inside the mineral sample, and of fractures, mineral distributions, fluid flow patterns, and other important phenomena inside the imaged geologic core. The data could be interpreted to yield fracture geometries, porosities, absolute and relative permeabilities, formation damage, fracture healing, flow through fractures, absorption and desorption, and other important behavior in which chemical and flow phenomena interact with major consequences for geologic sequestration. The approximate cost of the laboratory would be about \$3.5 million.

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Mineral Sequestration FY01 Annual Report

INTRODUCTION:

Increasing public concern related to the possible effects that anthropogenic emissions of CO₂ may have on global climate has led the Department of Energy (DOE) to embark on development of a number of carbon sequestration processes for mitigating carbon dioxide. Anthropogenic emissions of CO₂ generated by the combustion of fossil fuels are estimated at 6 GtC/year¹. The intent of the Carbon Sequestration program is to significantly decrease emissions of greenhouse gases and stabilize atmospheric CO₂ concentrations at 550 ppm by 2025. Complementary to traditional areas of energy research, such as improving energy efficiency or shifting to renewable or nuclear energy sources, carbon sequestration will allow continued use of fossil energy, buying decades of time needed for transitioning into less carbon-intensive and more energy-efficient methods for generating energy in the future.

Mineral carbonation, the reaction of CO₂ with non-carbonated minerals to form stable, benign mineral carbonates, has been identified as a possible safe, long-term, and unmonitored option for storing carbon dioxide²⁻⁷. In this chemical approach, CO₂ reacts with alkaline earth-containing silicate minerals, such as serpentine, Mg₃Si₂O₅(OH)₄, to form magnesite, MgCO₃. Moreover, Goff and Lackner have shown the economics of using magnesium silicates for carbonation and their abundance are well matched concerning the scale of CO₂ storage⁸. Current studies using serpentine, a Mg-rich, lamellar, hydroxide-based mineral, however, require prior pulverization of the mineral, removal of magnetite prior to carbonation, and thermal activation at high temperatures (630 °C) to successfully achieve rapid and high rates of conversion to magnesite. Thus, alternative chemical and mechanical methods focusing on surface activation of serpentine to accelerate the carbonation reaction efficiency have been under investigation^{9,10}.

SUMMARY ACCOMPLISHMENTS:

Presentations at American Chemical Society (ACS) National Meeting, San Diego, CA; First National Carbon Sequestration Conference, Washington, DC; and Pittsburgh Coal Conference, Newcastle, New South Wales, Australia

Daniel J. Fauth, an OST researcher, presented NETL's experimental results pertaining to mineral sequestration, a promising CO₂ sequestration technology option for converting anthropogenic CO₂ and magnesium silicates into permanent carbonate minerals, at the 222nd National Meeting of the American Chemical Society during April 2001. The manuscript was well received as determined by selection for nomination of the Robert Glenn award, a prestigious honor given by the Fuel Division, and the number of domestic inquiries with respect to NETL's mineral sequestration research. A second, more comprehensive manuscript entitled "Carbon Storage and Sequestration as Mineral Carbonates" was prepared and peer reviewed for outside publication.

An oral poster and manuscript entitled "Carbon Sequestration in the Office of Science and Technology at the National Energy Technology Laboratory" was given by Dr. White at the First National Carbon Sequestration Conference held in Washington, DC during May 2001. Current research projects, including CO₂ capture, geologic and oceanic sequestration of carbon dioxide, and advanced CO₂ conversion and reuse (*Mineral Sequestration*), were highlighted. The primary focus of NETL's Carbon Sequestration Science Program is to develop and evaluate, to the point of deployment, environmentally acceptable approaches to capture and geologically sequester CO₂.

Mr. Fauth co-authored a manuscript with M. Mercedes Maroto-Valer from the Pennsylvania State University's Department of Energy and Geo-Environmental Engineering and The Energy Institute entitled "Study of Magnesium Rich Minerals as Carbonation Feedstock Materials for CO₂ Sequestration." This work, conducted on serpentine, a Mg-rich, lamellar, hydroxide-based mineral, focused on surface characterization of serpentine with N₂ adsorption isotherms and scanning electron microscopy, along with investigations of economical chemical routes to increase its surface area. This work was presented at the 8th Annual Pittsburgh Coal Conference in December 2001.

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ACCOMPLISHMENTS:

An extensive review of the literature identified a number of academic and industrial laboratories that possess equipment and technology that may be used in the GSSF. Six laboratories were selected for site visits to examine relevant equipment and to hold discussions with laboratory personnel. Visited were: the USGS in Menlo Park, CA; Texas A&M University; MIT; University of Texas at Austin; Harvard University; and the New England Testing Lab, Boston, MA. Each of these laboratories has facilities suitable for use in the GSSF. For example, a vessel to house brine and sandstone samples under temperature and pressure conditions required to simulate sequestration conditions was observed at the labs. Each reactor vessel is capable, to some degree, of introducing brines in both static and dynamic flow conditions. Significantly, the following academic and industrial laboratories were identified as possessing the equipment and technology most readily able to simulate sequestration conditions as envisioned by the GSSF: Texas A&M University, MIT, and the New England Testing Lab.

To better understand the nature of the above three identified reactor vessels and the potential of each in addressing the needs of the GSSF, subsequent discussions were held with the chief researcher of each lab: Dr. Evans of MIT, Dr. Kronenberg of Texas A&M, and Dr. Martin of New England Testing Lab. The discussions centered around having each lab run tests on NETL-supplied brine and sandstone samples to better understand the potential of incorporating a specific reactor vessel in the GSSF. Both academic institutions required a long term, educational arrangement be entered into to perform any tests. Each academic institution envisioned the tests being done by a graduate student or a post-doctoral student funded and dedicated to the GSSF project. The industrial lab is able to perform tests on a fee basis alone. Given the time needed to implement a funded academic program, it is considered most expeditious to have the New England Lab perform initial tests on NETL samples to better understand the dynamics of incorporating instrumentation into the GSSF.

The nature of the GSSF is outlined in a flow chart which was completed as part of last year's work goals. The flow chart identifies the nine major data collection devices of the GSSF. The flow chart outlines in which of three test modes (pre-test mode, real-time test mode, or post-test mode) the instruments are utilized within the GSSF to obtain data on brine and sandstone samples. In part, the nature of the data obtained by the instrument and whether the data is obtained in a destructive or non-destructive manner dictates which mode the instrumentation is utilized within the GSSF. The online instrumentation included with the GSSF includes a gas chromatograph, an ion chromatograph, an ICP, a laser scanning confocal microscope, an AA, an SEM, a petrographic microscope, computer-aided tomography, a magnetic resonance imaging device, an electron microprobe, and an X-ray diffractometer.